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With international search report.

(54) Title: FIRE-RESISTANT FIBRE CEMENT PRODUCTS

(57) Abstract

An autoclaved fibre cement product comprising, cellulose fibres, cementitious material, and 1 % to about 40 % of magnesite on a dry weight basis. In preferred embodiments the percentage of cellulose fibres is in the range from 7 % to 8.5 % and the magnesite in a preferred range of between 25 % to 40 %. Silica and portland cement may also be included.

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Fire-resistant fibre cement products

TECHNICAL FIELD

The present invention relates to fibre-reinforced cementitious products and in particular to fire resistant fibre cement products.

BACKGROUND ART

The invention has been developed primarily for production of fire-resistant autoclaved-fibre cement boards for both internal and external applications and will be described hereinafter with reference to this use. However, it will be appreciated that the invention is not limited to this particular field.

In order to achieve a given fire rating, it is necessary that the fibre cement boards comply with the relevant Industrial Standards applicable to the country in which they are to be used. Whilst the standards vary from country to country, the criteria by which the materials are evaluated are much the same. The main criteria can be summarised as:

(a) Combustibility (temperature rise/flaming criterion)

(b) Thermal Shrinkage

(c) Heat and Smoke Evolved

The existing fibre/cement boards available in Australia do not, for example, meet the requirements of the local combustibility Standard (AS1530 part 1) on the basis that they fail the flaming criterion. By contrast, the same boards fail the temperature rise criterion of the equivalent Japanese Standard JIS1321.

In addition to meeting the requirements of the relevant, fire related Industrial Standards, the boards also need to have certain desired mechanical and insulating properties.

It is an object of the present invention to provide an improved fibre cement product which will avoid or at least ameliorate the disadvantages of the prior art and thereby provide a product of improved combustibility rating.

The invention proceeds from the discovery that the inclusion of Magnesite (Magnesium Carbonate) in a fibre/cement board, improves the combustibility rating characteristics of the material thereby rendering it more fire resistant, whilst at least retaining, and in some cases improving, the required mechanical and structural characteristics of the product.

DISCLOSURE OF INVENTION

According to the invention there is provided an autoclaved fibre cement product comprising, cellulose fibres, cementitious material, and 1% to about 40% of Magnesite.

In a preferred embodiment this product also

includes silica.

Preferably the proportion of cellulose fibres is in the range from 7% to 8.5%.

Preferably also the proportion of magnesite is between 25% to 40%.

It has been found that fibre cement mixtures according to this invention, that is incorporating 1% to 40% magnesite, display improved fire resistance qualities.

All percentage inclusion levels herein described refer to the percentage on a dry weight basis prior to processing.

More particularly tests have shown that pressed and unpressed autoclaved fibre cement mixtures made according to the invention with 8.5% cellulose demonstrate the
following advantages:

(I) The invented product has an improved combustibility rating when tested compared to normal mixes with less than 5% cellulose which performed better in these tests than normal mixes containing 7 to 8.5% cellulose.

(II) The product demonstrates reduced thermal shrinkage when tested and compared with normal mixes containing 7 or 8% cellulose.

(III) The product, when tested, is found to have an equal modulus of rupture, an equal modulus of elasticity

and an equal tensile strength to normal mixes containing 7 or 8% cellulose.

(IV) The invented product also demonstrates an equal tensile strain to failure to normal mixes containing 7 or 8% cellulose.

(V) The invented product has a 100% greater flexural strain to failure than normal mixes containing 7 or 8% cellulose.

(VI) The invented product, when tested, demonstrates only 75% precarbonation moisture movement of that of normal mixes containing 7 or 8% cellulose.

(VII) The invented product has a cracking resistance index up to 500% above normal mixes containing 7 or 8% cellulose.

(VIII) The invented product has improved fire insulation characteristics compared to normal mixes containing 7 or 8% cellulose.

(IX) The invented product demonstrates an improved general fire performance which may be expected to continue improving after ageing and natural carbonation.

The following examples served to illustrate, without limiting the invention.

BRIEF DESCRIPTION OF DRAWINGS

Figure 1 is a graph showing the variation in temperature with time during combustibility tests for specimens containing 8.5% cellulose and various percentages

of magnesite.

Figure 2 is a graph showing the variation of maximum temperature and time to maximum temperature with percentage substitution of magnesite.

Figure 3 is a graph showing the actual and predicted variation of percentage shrinkage with percentage magnesite content for specimens having 8.5% and 5% cellulose content.

BEST MODE FOR CARRYING OUT THE INVENTION

COMBUSTIBILITY INVESTIGATIONS

Magnesite was incorporated in various proportions into hand made specimens containing 8.5% cellulose and tested in accordance with JIS1321. The resulting time-temperature curves are shown in Figure 1.

The addition of magnesite was found not only to reduce the peak exotherm temperature but also to increase the time at which the exotherm occurs. This is exemplified by the following table and graph shown in Figure 2.

Percentage Addition Magnesite	Peak Temperature °C	Time to Peak Temp	Time to 800°C
0	826	390	205
5	822	430	209
10	821	374	224
20	811	390	295
30	814	450	325
40	802	415	375

From the results above it can be seen that the substitution of silica by between 30 and 40% of magnesite is necessary to pass the JIS test for combustibility when used in a formulation containing

8.5% cellulose.

The mechanism by which the magnesite reduces the peak temperature is apparent in the time-temperature curves of the high percentage magnesite samples shown in Figure 1, where there is a pronounced dip in the curve around 700°C. This is due to the decomposition of magnesite which commences around 450°C. The dip in the time-temperature curve occurs at a higher apparent temperature due to the thermal lag arising from the location of the thermocouple.

THERMAL SHRINKAGE

Investigation on the laboratory bench scale of the thermal shrinkage of mixes containing varying proportions of cellulose and Magnesite gave the results listed in the following table.

% Cellulose	% Magnesite	% Shrinkage	
		Actual	Predicted
5.00	0.00	5.70	6.0
5.00	10.00	5.00	5.1
5.00	20.00	4.00	4.2
5.00	30.00	3.80	3.3
8.50	0.00	7.20	7.4
8.50	5.00	7.10	6.9
8.50	10.00	6.90	6.5
8.50	20.00	5.70	5.6
8.50	30.00	4.10	4.7
8.50	40.00	3.70	3.8

Analysis of the results indicated that the following relationship appears to apply the thermal shrinkage of Magnesite containing materials.

$$Shr\ \% = 4 + 0.39*\%Cel - 0.09*\%Mag$$

The predicted and actual results are shown in Figure 3.

MECHANICAL PROPERTIES

In light of the above discoveries an investigation was undertaken using a pilot scale Hatschek Machine and the following mixes were evaluated.

Percentage For Mix Number

Component	1	2	3	4
Cellulose	7.0	7.0	8.0	8.0
Cement	35.5	35.5	35.1	35.1
Silica	17.6	12.6	25.7	20.7
Magnesite	35.0	40.0	27.5	32.5
Filler*	4.3	4.3	4.3	4.3

* Selected from the group comprising mineral oxides, hydroxides and clays.

Each mix was evaluated for tensile and flexural mechanical properties, moisture movement and durability before and after carbonation. Tests were also carried out for combustibility and thermal shrinkage at 1000° before and after carbonation. Pilot fire tests were carried out using the material in single lamination of 5 mm thickness and double and treble laminations, at 10 and 15 mm thicknesses respectively.

Results of these tests were evaluated against the known average properties of standard fibre cement sheet without the addition of Magnesite. The following are typical results for uncarbonated sheets compared to standard formulation with 8 % Cellulose in the equilibrium condition.

Property	Unpressed	Pressed
Modulus of Rupture	Equal	Equal
Modulus of Elasticity	Equal	Equal
Strain to Failure	+100%	+50%
Interlaminar Bond	80%	80%
Tensile Strength	Equal	Equal
Tensile Strain to Failure	Equal	Equal
Moisture Movement Precarbonation	75%	75%
Cracking Resistance Index	up to +500%	up to +200%

In all properties except interlaminar bond the properties of the Magnesite substituted sheets containing either 7 or 8% Cellulose exceeded those of the unsubstituted sheets contain 8% cellulose. In general plain sheets containing only 7% cellulose have poorer mechanical properties than sheets containing 8% cellulose therefore the substitution of Silica by Magnesite represents a significant advantage.

Of particular significance is the increase in the Cracking Resistance Index. This is a measure of the propensity for the sheet to crack when subject to shrinkage while restrained. This property was improved by a factor of up to 6 over the plain board.

THERMAL SHRINKAGE

The preliminary investigation predicted that the shrinkage of unpressed and uncarbonated Magnesite

substituted sheets should lie between 3.5 and 4.3% depending on the mix. The following results were obtained.

Mix	Predicted Shrinkage	Actual Shrinkage
1	4.26	2.3
2	3.81	3.0
3	3.97	2.6
4	3.52	2.4

Additionally it was found that the pressing the sheets reduced the shrinkage by an average of 0.4% while carbonating the sheets increased it marginally by about 0.06%. Pressing and carbonating the sheet reduced the shrinkage by about 0.2%. This shows that enhanced resistance against warping in a fire should be expected with compressed sheet compared to the unpressed sheets. Additionally there should not be any diminishment in performance with exposure to natural weathering.

PILOT FIRE TESTS

The pilot fire tests showed some inconsistencies. The following results were obtained for insulation value of a single 5 mm layer of Magnesite substituted material.

Mix	Insulation Value (minutes)
1	6.5
2	3.5
3	8.5
4	3.0
Plain mix*	3.5

* Mix containing 7% Cellulose.

It appears that the too great a substitution of Silica by Magnesite is not beneficial and that the best results may be obtained with less than the optimum amounts for shrinkage. It is speculated that the insulation value may be reduced because the evolution of CO_2 from the Magnesite during the fire test may transfer heat through the sheet by mass diffusion. This effect is compensated to some extent by the heat required to cause the decomposition of the Magnesite thus there will be a changeover percentage where the apparent conductivity to heat increases with increase in Magnesite content.

Therefore, from the results of the tests conducted to date, it appears that in order to improve the combustibility rating of the product whilst simultaneously achieving acceptable insulation values and general mechanical and structural characteristics, a product incorporating from around 25% to about 40% Magnesite is preferred.

Although the invention has been described with reference to only selected examples, it will be appreciated by those skilled in the art that the invention may be embodied in many other forms including products not including silica and products containing lesser amounts of cellulose.

CLAIMS

1. An autoclaved fibre cement product comprising, cellulose fibres, cementitious material, and 1% to about 40% of magnesite on a dry weight basis.

2. An autoclaved fibre cement product according to claim 1 wherein the proportion of cellulose fibres is in the range from 7% to 8.5% on a dry weight basis.

3. An autoclaved fibre cement product according to any one of the preceding claims wherein the proportion of magnesite is between 25% to 40% on a dry weight basis.

4. An autoclaved fibre cement product according to any one of the preceding claims including about 35% portland cement on a dry weight basis.

5. An autoclaved fibre cement product according to any one of the preceding claims including silica.

6. An autoclaved fibre cement product according to claim 5 wherein the proportion of silica is in the range of 12% to 25% on a dry weight basis.

7. An autoclaved fibre cement product according to any one of the preceding claims including about 4% filler material wherein the filler material is selected from the group comprising mineral oxides, hydroxides and clays.

8. An autoclaved fibre cement product substantially as herein described with reference to the examples.

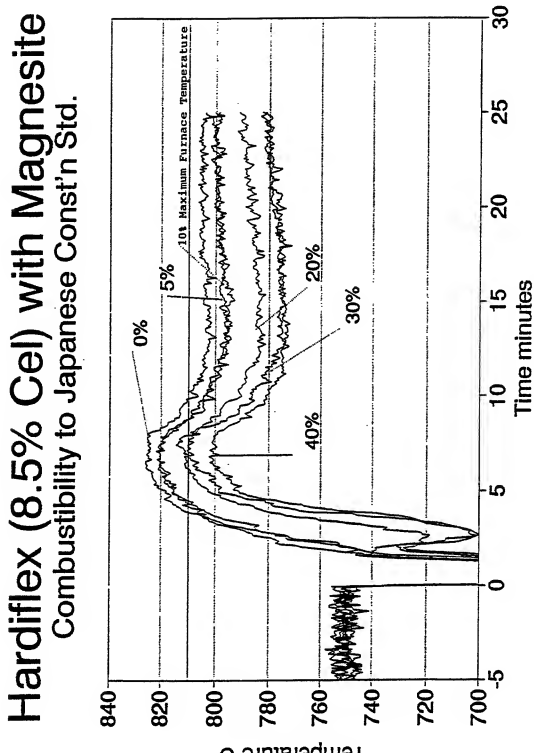
Hardiflex (8.5% Cel) with Magnesite
Combustibility to Japanese Const'n Std.
101 Maximum Furnace Temperature
0%
5%
20%
30%
40%
Temperature C
840
820
800
780
760
740
720
700
-5
0
5
10
15
20
25
30
Time minutes

FIG. 1

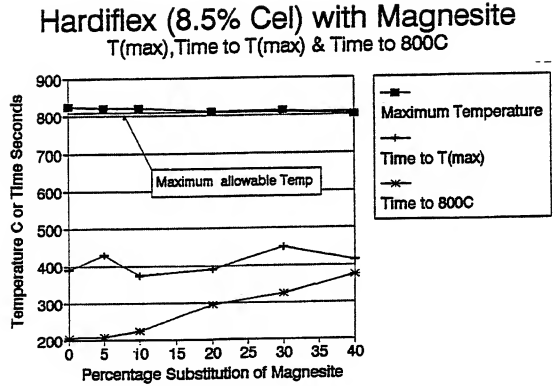
Hardiflex (8.5% Cel) with Magnesite
T(max),Time to T(max) & Time to 800C
Temperature C or Time Seconds
900
800
700
600
500
400
300
200
0 5 10 15 20 25 30 35 40
Percentage Substitution of Magnesite
Maximum allowable Temp
Maximum Temperature
Time to T(max)
Time to 800C

FIG. 2

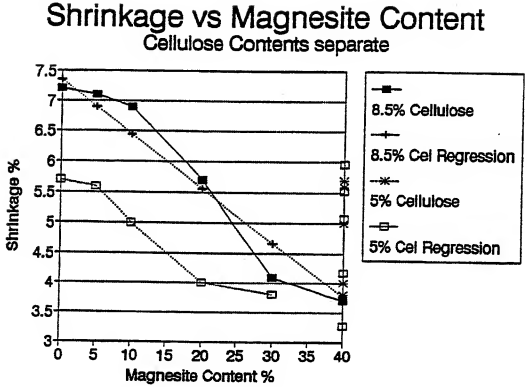
Shrinkage vs Magnesite Content
Cellulose Contents separate
Shrinkage %
7.5
7
6.5
6
5.5
5
4.5
4
3.5
3
0 5 10 15 20 25 30 35 40
Magnesite Content %
8.5% Cellulose
8.5% Cel Regression
5% Cellulose
5% Cel Regression

FIG. 3

INTERNATIONAL SEARCH REPORT

International Application No. PCT/AU 91/00271

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) 6

According to International Patent Classification (IPC) or to both National Classification and IPC

Int. Cl.[5] C04B 14/26, 16/02, 28/04, E04B 1/14

II. FIELDS SEARCHED

Minimum Documentation Searched 7

Classification System	Classification Symbols
IPC	C04B 14/26, 16/02, 31/14, 31/36

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched 8

AU: IPC as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT 9

Category*	Citation of Document,[11] with indication,[12] where appropriate, of the relevant passages	Relevant to Claim No 13
Y	Chemical Abstracts, Volume 96, no. 18, 3 May 1982, page 339, abstract no. 96:147952c, JP,A, 56-169167 (ASAHI GLASS) 25 December 1981 (25.12.81)	1-8
Y	Chemical Abstracts, Volume 98, no. 22, 30 May 1983, page 310, abstract no. 98:184598d, JP,A, 57-200253 (IKEDA, TAKASHI) 8 December 1982 (08.12.82)	1
Y	Chemtech, Volume 18, no. 8, August 1988 (Washington D C), A A Moslemi, "Inorganically Bonded Wood Composites" pages 504-510	1
A	Chemical Abstracts, Volume 100, no. 16, 16 April 1984, page 295, abstract no. 100:125952b, IL,A, 62790 (FRIEDMANN, PESSAH) 31 July 1983 (31.07.83)	
A	Chemical Abstracts, Volume 105, no. 4, 28 July 1986, page 303, abstract no. 105:28878c, JP,A, 60-180974 (LIN, JUI CHE) 14 September 1985 (14.09.85)	
P,A	Chemical Abstracts, Volume 113, no. 22, 26 November 1990, page 335,	

* Special categories of cited documents: 10

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
22 August 1991 (22.08.91)	29 August 91
International Searching Authority	Signature of Authorized Officer
Australian Patent Office	M BREMERS

Form PCT/ISA/210 (second sheet) (January 1985)

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category*	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
	abstract no. 113:196727m, JP,A, 02-157148 (ASAHI GLASS) 15 June 1990 (15.06.90)	
A	Patent Abstracts of Japan, C-755, page 160, JP,A, 02-157148 (ASAHI GLASS) 15 June 1990 (15.06.90)	

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

V.[] OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE 1

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. [] Claim numbers ..., because they relate to subject matter not required to be searched by this Authority, namely:

2. [] Claim numbers ..., because they relate to parts of the international application that do comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. [] Claim numbers ..., because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4 (a):

VI.[] OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING 2

This International Searching Authority found multiple inventions in this international application as follows:

1. [] As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.
2. [] As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. [] No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. [] As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

[] The additional search fees were accompanied by applicant's protest.
[] No protest accompanied the payment of additional search fees.

DERWENT-ACC-NO: 1992-056768

DERWENT-WEEK: 199213

TITLE: Fireproof autoclaved cellulose fibre-cement prods. comprising cellulose fibres, cementitious material and magnesite

INVENTOR: COOKE A M; HODGSON W B ; SLOANE B P

PATENT-ASSIGNEE: HARDIE J & CO PTY L[HARDN]

PRIORITY-DATA: 1990AU-001110 (July 10, 1990)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE
WO 9200927 A	January 23, 1992	EN
AU 9175366 A	January 16, 1992	EN

DESIGNATED-STATES: JP US AT BE CH DE DK ES FR GB GR IT LU NL SE

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
WO1992000927A	N/A	1991WO-AU00271	June 26, 1991
AU 9175366A	N/A	1990AU-001110	July 10, 1990

INT-CL-CURRENT:

TYPE	IPC DATE
CIPS	C04B14/26 20060101
CIPS	C04B16/02 20060101
CIPS	C04B28/02 20060101

CIPS C04B28/04 20060101
CIPS E04B1/14 20060101

ABSTRACTED-PUB-NO: WO 9200927 A

BASIC-ABSTRACT:

An autoclaved fibre cement prod. contg. cellulose fibre, cementitious material and 1-40 wt.% magnesite (magnesium carbonate). The prod. may also contain silica, Portland cement and a filler such as (hydr)oxides and clays. Pref. 7-8.5 wt.% fibre, 25-40 wt.% magnesite. 12-25 wt.% silica. 35 wt.% Portland cement. 4 wt.% filler.

USE/ADVANTAGE - Fibre cement boards made from this cement are fire resistant, show reduced thermal shrinkage, greater flexural strain and resistance to cracking.

TITLE-TERMS: FIRE AUTOCLAVE CELLULOSE FIBRE CEMENT PRODUCT COMPRISE CEMENTED MATERIAL MAGNESITE

DERWENT-CLASS: L02 Q43

CPI-CODES: L02-C; L02-D15A;

UNLINKED-DERWENT-REGISTRY-NUMBERS: ; 1694U

SECONDARY-ACC-NO:
CPI Secondary Accession Numbers: 1992-025607
Non-CPI Secondary Accession Numbers: 1992-043229